

IN THE UNITED STATES  
PATENT AND TRADEMARK OFFICE

#9

**Patent Application**

Inventors: Mary L. Mandich  
William D. Reents

Case No.: 9-10

Serial No.: 09/912,129

Group Art Unit: 1731

Filing Date: July 24, 2001

Examiner: John M. Hoffman

Title: PROCESS FOR FABRICATING OPTICAL FIBER

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DECLARATION UNDER 37 C.F.R. 1.132

1. I, Mary L. Mandich, received a Ph.D. in chemistry from Columbia University. I have been employed at Bell Laboratories since 1983 and have at least 5 years of experience in technologies relating to optical fiber fabrication. These technologies include sol-gel fabrication and manufacture of optical fiber preforms. I am a co-inventor on the above-referenced patent application.

2. I, William D. Reents, Jr., received a Ph.D. in chemistry from Purdue University. I have been employed at Bell Laboratories since 1980 and have at least 8 years of experience in technologies relating to optical fiber fabrication. These technologies include sol-gel fabrication and manufacture of optical fiber preforms. I am a co-inventor on the above-referenced patent application.

3. I am familiar with U.S. Patent No. 5,356,447 by Bhandarkar (Herein, referred to as "Bhandarkar") and U.S. Patent No. 4,264,347 by Shintani et al (Herein, referred to as "Shintani").

4. Bhandarkar describes processes for removing refractory particles from an unsintered preform body. See, e.g., Bhandarkar, col. 1, line 64, to col. 2, line 2. Bhandarkar's processes include exposing the preform body to a gaseous mixture containing a halogenated compound to cause a reaction between refractory oxide

particles in the preform body and the halogenated compound. See, Bhandarkar, claim 1. Exemplary of the halogenated compounds used in the processes described by Bhandarkar is thionyl chloride.

5. Bhandarkar describes a functionality of his halogenated compounds that is important to his method. The functionality described by Bhandarkar is that the halogenated compounds react with refractory particles to liberate gaseous byproducts. See, Bhandarkar, claim 1. The liberation of a gaseous byproduct reduces the size of the refractory particles during performance of the method of Bhandarkar. *Id.* Thus, the functionality that Bhandarkar describes is important to his use of halogenated compounds for gas removal of refractory particles.

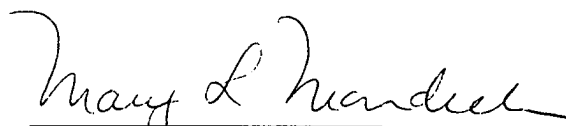
6. Shintani describes processes for removing imperfections at interfaces between the glass rod and tube joined in the rod-in-tube method for making an optical fiber preform. Shintani's processes include exposing such glass bodies to chemical agents that surface treat the bodies. Shintani, col. 5, lines 46 –61. Shintani teaches that the chemical agents have a functionality important to the rod-in-tube method. The functionality is that the chemical agents modify a surface glass layer of a glass body so that, at temperatures used in the rod-in-tube method, the modified surface glass layer has a lower viscosity and also has an increased affinity for other surface glass layers modified by the chemical agents. *Id.* Shintani teaches that this functionality enables treatments with his chemical agents to produce interfaces free of voids when the treated surfaces are joined, e.g., during the rod-in-tube methods for fabrication of optical fiber preforms. *Id.*

7. The surface treatment functionality that Shintani describes for his chemical agents is not equivalent to the reaction functionality described by Bhandarkar. One reason that the above described functionalities are inequivalent is that the two functionalities describe production of different types byproducts. The reaction functionality described by Bhandarkar produces gaseous byproducts at temperatures for the Bhandarkar process whereas the surface treatment functionality described by Shintani produces viscous surface layers, e.g., liquids, at relevant temperatures for the rod-in-tube method.

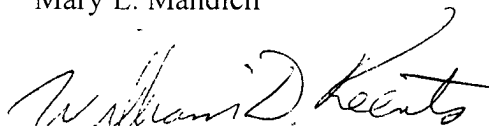
8. One of skill in the art of manufacturing optical fiber preforms would not find an agent functionality for generating viscous glass byproducts to be equivalent to a reaction functionality for generating gaseous byproducts. This difference in byproduct production functionalities that Shintani and Bhandarkar attribute to their respective surface treatment agents and halogenated compounds would not normally convince one of skill in the art that the agents of Shintani could be interchanged for the halogenated compounds used in the Bhandarkar process. For this reason, I believe that Shintani's teaching that non-oxygenated sulfur halides could be used to surface treat glass bodies would not have made the use of such compounds in Bhandarkar's process for gas removal of refractory particles obvious to one of skill in the art.

9. Herein, I certify that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true. I also understand that willful false statements and the like are punishable by fine, imprisonment or both under 18 U.S.C. 1001 and that willful false statements and the like may jeopardize the validity of the application-at-issue or any patent issuing thereon.

Date: 04/22/02

  
Mary L. Mandich

Date: April 22, 2002

  
William D. Reents

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IN THE UNITED STATES  
PATENT AND TRADEMARK OFFICE

**PATENT APPLICATION**

Inventors: Mary Louise Mandich  
William David Reents

Case No. 6-7

Title Process For Fabricating Optical Fiber

ASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D. C. 20231

COPY OF PAPERS  
ORIGINALLY FILED

SIR:

DECLARATION UNDER 37 C.F.R. § 1.132

1. I, Mary L. Mandich, received a Ph.D. in Chemistry from Columbia University. I have been employed at Bell Laboratories since 1983, and have at least 5 years of experience in technologies relating to optical fiber fabrication, including sol-gel technology and preform manufacture. I am a coinventor of the above-captioned patent application.

2. I, William D. Reents, Jr., received a Ph.D. in Chemistry from Purdue University. I have been employed at Bell Laboratories since 1980, and have at least 8 years of experience in technologies relating to optical fiber fabrication, including sol-gel technology and preform manufacture. I am also a coinventor of the above-captioned patent application.

3. I am familiar with the references cited in what I understand to be the final Office Action in my parent application, specifically U.S. Patent No. 4,969,941 to Kyoto et al. ("Kyoto"), U.S. Patent No. 5,356,447 to Bhandarkar ("Bhandarkar"), and U.S. Patent No. 4,264,347 to Shintani et al. ("Shintani").

4. I have considered the Kyoto reference. It is clear to me that Kyoto discusses only soot bodies formed by VAD (vapor axial deposition) or

OVD (outside vapor deposition) processes. For example, I note the discussion at Col. 1, lines 18 to 56, which relates solely to VAD. I also note the discussion at Col. 7, lines 18 to 32, which would indicate to someone skilled in the art of fiber preforms that Kyoto is referring to VAD or OVD bodies. I further note the discussion at Col. 16, lines 16 to 37, which also would indicate a VAD or OVD body.

5. These VAD or OVD soot bodies of Kyoto are not sol-gel bodies, e.g., bodies made according to a process such as disclosed in U.S. Patent No. 5,240,488, which I note is discussed in the Background section of my patent application. No one skilled in the art of fiber preform manufacture would consider these soot bodies to be sol-gel bodies.

6. One skilled in the art of fiber preform manufacture is aware that VAD and OVD bodies, such as discussed in Kyoto, typically do not contain a measurable amount of refractory metal oxide particles, unlike sol-gel bodies. Specifically, the gases used in VAD and OVD processes to form a glass body (e.g.,  $\text{SiCl}_4$ ) are of such high purity that substantially no refractory metal oxide particles exist in the formed body.

7. By contrast, sol-gel bodies typically start with a silica particle dispersion. Due to contamination present in the initial silica dispersion and introduced by both mixing and molding equipment, the presence of refractory metal oxide particles in the formed sol-gel body is typically unavoidable. This contamination is discussed in the Background section of the patent application at page 3, lines 13 to 15.

8. I note that Shintani discloses a step of exposing the sintered silica body to hydrofluoric acid prior to gas treatment, in order to clean the surface of the sintered body, e.g., at Col. 4, lines 13-15. This HF cleaning step could not be performed on a porous glass body, e.g., a pre-sintered sol-gel body of the type discussed in Bhandarkar, without substantially destroying the porous glass body.

9. It was discovered subsequent to filing my parent application that the effectiveness of thionyl chloride ( $\text{SOCl}_2$ ) is highly dependent on the furnace configuration, the type of gas flow over the body in the furnace, the loading of the furnace, etc. Some of the data presented in the application regarding the effectiveness of thionyl chloride in etching refractory metal oxide particles was obtained from different furnaces, and the results therefore are not necessarily illustrative of the advantages of the invention. Specifically, the experiments reported in Tables 1 through 4 for 17% thionyl chloride were performed in a different furnace than the 18% thionyl chloride runs reported in Tables 2 and 4, which explains the differing results.

10. The reasons for thionyl chloride's high sensitivity appears to be related to its slow decomposition. As reflected in the table below, small changes in residence time of thionyl chloride within a furnace can have significant effects on the concentration, due to this slow decomposition. (See, e.g., the significant difference between 4 minutes and 5 minutes.) Thus, the particular furnace configuration, gas flow, furnace loading, etc. will affect the concentration of thionyl chloride that reaches a particular body, and will thereby effect the extent of particle etching performed.

**Volume percent of thionyl chloride delivered as measured by mass spectrometry, versus residence time of the thionyl chloride in the same furnace**

Measured Vol.% Thionyl Chloride	Residence time (minutes)
20	4
10	5
9.2	40
7.8	160

My experience with thionyl chloride, consistent with the above discussion, has clearly indicated that the compound is highly sensitive to a variety of treatment conditions, this sensitivity creating the potential for interference with the desired etching of refractory metal oxide particles. By contrast, I

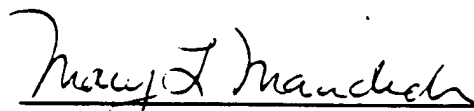

hav neither observed nor am aware of any such sensitivity in sulfur chloride treatments of sol-gel bodies.

11. Becaus of this sensitivity of thionyl chloride, a meaningful comparison of thionyl chloride etching rates to sulfur chloride etching rates can only be made where the experiments were performed under the same furnace conditions. In the application, such a meaningful comparison is found in Tables 2 and 4, e.g., for the 6.4% sulfur monochloride vs. the 18% thionyl chloride, which were tested under the same furnace conditions. (The data of Tables 1 and 3 does not include thionyl chloride runs performed in the same furnace as sulfur chloride runs.) The Tables clearly show that the sulfur monochloride is equally effective as thionyl chloride, but at nearly 1/3 the concentration, which is a major advantage in a commercial process. In addition, sulfur dichloride tested at a concentration of 3.2% and a temperature of 650°C, in the same furnace and according with the same techniques described in the application, exhibited a volume normalized etch rate for zirconia (see Table 4) of about 60 moles/hour. Thus, the sulfur dichloride is even more efficient, i.e., able to provide about the same etching rate with half the concentration of sulfur monochloride and nearly 1/6 the concentration of thionyl chloride.

12. All statements made herein based on my own knowledge are true, and statements made on information and belief are believed to be true. I understand that willful false statements and the like are punishable by fine and/or imprisonment (18 U.S.C. § 1001), and may jeopardize the validity of the present application and any patents issuing from the present application.

Date: 12-29-99

Date: 12-9-99

  
Mary L. Mandich  
  
William D. Reents, Jr.